

# Evidence for Triplet Superconductivity in a Superconductor-Ferromagnet Spin Valve

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We have studied the dependence of the superconducting (SC) transition temperature on the mutual orientation of magnetizations of Fe1 and Fe2 layers in the spin valve system CoO<sub>x</sub>/Fe1/Cu/Fe2/Pb. We find that this dependence is nonmonotonic when passing from the parallel to the antiparallel case and reveals a distinct minimum near the orthogonal configuration. The analysis of the data in the framework of the SC triplet spin valve theory gives direct evidence for the long-range triplet superconductivity arising due to noncollinearity of the two magnetizations.

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Recent theories predict that *spin-triplet* components of the superconducting (SC) condensate can be generated under certain conditions in heterostructures comprising a conventional superconductor (S) and a ferromagnet (F) (for a review see, e.g., [1]). The components with the spin projections (upon exchange field)  $S_z = \pm 1$  can then penetrate into a ferromagnet over a long distance. The appearance of the triplet components can be understood in the framework of the picture proposed by Demler *et al.* [2]: Upon entering the F layer, a pair of electrons with spin up and spin down  $|\uparrow\downarrow\rangle$  acquires a center-of-mass momentum  $Q = 2\hbar/v_F$ , since the exchange field acts on two electrons as a potential of opposite signs. Here,  $\hbar$  is the exchange field and  $v_F$  is the Fermi velocity. Taking into account a complementary pair with opposite spins,  $|\downarrow\uparrow\rangle$  (necessary for fermionic antisymmetry), that acquires the opposite center-of-mass momentum  $-Q$ , one combines them into a singlet Cooper pair. The wave function of this Cooper pair becomes spatially modulated in the F layer as  $\cos(Qx)$ , where  $x$  is the center-of-mass coordinate. This is the reason for oscillating behavior of the S/F proximity effect [3]. However, at that time, Demler *et al.* did not pay attention to the triplet component which inevitably arises in the framework of the same approach. Indeed, the singlet state of the two spins (corresponding to the singlet Cooper pair) is an antisymmetric combination  $|\uparrow\downarrow\rangle - |\downarrow\uparrow\rangle$  (hereafter, we omit the normalizing factor  $1/\sqrt{2}$  for brevity). In the F layer, these two terms acquire oscillating exponential prefactors differing by the sign of  $Q$ . This yields [4]

$$e^{iQx}|\uparrow\downarrow\rangle - e^{-iQx}|\downarrow\uparrow\rangle = (|\uparrow\downarrow\rangle - |\downarrow\uparrow\rangle)\cos Qx + (|\uparrow\downarrow\rangle + |\downarrow\uparrow\rangle)i\sin Qx, \quad (1)$$

where the cosine term is again the singlet component, whereas the sine term is the triplet component with zero  $z$  projection (in the direction of the exchange field). If we

now introduce another *noncollinear* direction of  $\hbar$ , we can rotate the quantization axis accordingly, and then in the new coordinate system we immediately obtain also the components with  $S_z = \pm 1$  (i.e.,  $|\uparrow\uparrow\rangle$  and  $|\downarrow\downarrow\rangle$ ). Those components are not destroyed by the exchange field and can penetrate deeply into the F layer just as the singlet superconductivity penetrates into a nonmagnetic metallic layer; therefore, they are often referenced as “long-range” triplet components (LRTC).

In this Letter, we present experimental evidence for the occurrence of the LRTC of the SC condensate in the multilayer spin-valve heterostructure CoO/Fe1/Cu/Fe2/Pb. It is manifested in the nonmonotonic behavior of the SC transition temperature  $T_c$  of the Pb layer upon gradual rotation of the magnetization of the ferromagnetic Fe2 layer  $\mathbf{M}_{\text{Fe2}}$  with respect to the magnetization of the Fe1 layer  $\mathbf{M}_{\text{Fe1}}$  from the parallel (P) to the antiparallel (AP) orientation. We observe a clear minimum of  $T_c$  for the orthogonal configuration of  $\mathbf{M}_{\text{Fe1}}$  and  $\mathbf{M}_{\text{Fe2}}$ . As follows from our analysis in the framework of the theory of the SC triplet spin valve [5], such minimum of  $T_c$  is a fingerprint of the spin-triplet component generated by noncollinear magnetizations  $\mathbf{M}_{\text{Fe1}}$  and  $\mathbf{M}_{\text{Fe2}}$  [6].

The suppression of  $T_c$  in the S layer of an S/F1/F2 proximity system studied in our work takes place due to “leakage” of Cooper pairs into the F part. In this language, the generation of the LRTC at noncollinear magnetizations opens up an additional channel for this leakage, hence  $T_c$  should be suppressed stronger. Note that the triplet superconducting correlations are generated from the singlet ones (conversion due to the exchange field), reducing the amplitude of the singlet component in the S layer and thus “draining the source” of superconductivity in the whole system [7]. This effect is substantial since the magnitudes of the proximity-induced singlet component and the LRTC can be of the

same order near the interface of the S layer (if the thickness of the adjacent F layer is smaller than its coherence length).

Similarly to the S/F1/F2 system, the LTRC due to noncollinear ferromagnets is also generated in a multilayer of the F1/S/F2 type, studied both theoretically and experimentally [8–10]. At first sight, the same arguments about possibility to additionally suppress  $T_c$  due to leakage of Cooper pairs into the long-range triplet channel are also valid here. However, this suppression is masked by a simple effect of (partial) mutual compensation of the exchange fields of the two F layers at nonparallel orientations. This effect turns out to be the main one in the F1/S/F2 geometry, and its monotonicity leads to a monotonic  $T_c(\alpha)$  dependence [8–10]. On the other hand, in the S/F1/F2 geometry the outer F2 layer is separated from the S layer, and the mutual compensation of exchange fields has a much weaker effect on the latter. It is this feature that makes the S/F1/F2 structure advantageous for investigating a nontrivial influence of the triplet component on  $T_c(\alpha)$ .

The basis of the present work has been formed by our earlier studies of the SC spin valve effect in the multilayer system CoO/Fe1/Cu/Fe2/In [11]. In addition to the observation of the full spin valve effect we have found that its magnitude  $\Delta T_c = T_c^{\text{AP}} - T_c^{\text{P}}$ , i.e., the difference of  $T_c$  values for the AP and P mutual orientations of the magnetizations of the Fe1 and Fe2 layers, shows an oscillating sign-changing behavior as a function of the thickness of the Fe2 layer due to the interference effect of the SC pairing function in the Fe2 layer [12, 13]. As discussed above, the activation of the triplet channel should be visible in an additional suppression of  $T_c$  for noncollinear arrangements of  $\mathbf{M}_{\text{Fe1}}$  and  $\mathbf{M}_{\text{Fe2}}$ . Unfortunately, such experiment on the CoO/Fe1/Cu/Fe2/In system turned out to be unrealizable under well-controlled conditions owing to a low value of  $T_c$  for indium and its extreme sensitivity to small out-of-plane tilting of the external magnetic field. In this respect, lead has much better SC critical parameters, which has determined its choice as an S layer in the present work.

The CoO/Fe1/Cu/Fe2/Pb samples were prepared by classical electron beam evaporation in an ultra-high vacuum chamber (for details see [13]). In order to obtain a continuous Pb layer with the resistivity ratio  $RRR = R(300\text{ K})/R(10\text{ K}) > 10$  [where  $R(300\text{ K})$  and  $R(10\text{ K})$  are the electrical resistivity of samples at 300 and 10 K, respectively], the evaporation rate for the Pb layer was set to 3 nm/s [14]. The easy axis of the magnetization which is induced by residual magnetic field in our vacuum system was directed parallel to the long side of our rectangular-shaped samples.

We have studied six spin valve samples CoO<sub>x</sub>(4 nm)/Fe(2.5 nm)/Cu(4 nm)/Fe( $d_{\text{Fe2}}$ )/Pb(35 nm) with the thickness of the Fe2 layer  $d_{\text{Fe2}} = 0.6, 0.7, 0.9, 1.0, 1.3, 1.5\text{ nm}$  and one reference sam-

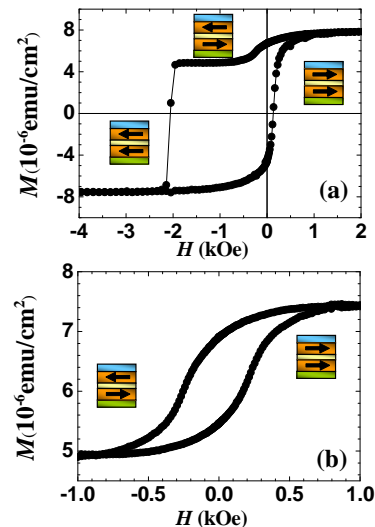


FIG. 1. (Color online) (a) Major magnetic hysteresis loop for the CoO/Fe1/Cu/Fe2/Pb sample with  $d_{\text{Fe2}} = 0.7\text{ nm}$  cooled from room temperature down to  $T = 4\text{ K}$  in a magnetic field  $H = 4\text{ kOe}$ ; (b) central part of the minor hysteresis loop for the same sample due to the reversal of the magnetization of the free Fe2 layer.

ple CoO<sub>x</sub>(4 nm)/Cu(7 nm)/Fe(1 nm)/Pb(35 nm)/SiN<sub>x</sub>(85 nm). The CoO<sub>x</sub> layer was used to pin the magnetization of the adjacent Fe layer (Fe1), whereas the role of the Cu layer is to decouple the magnetization of the Fe2 layer from that of the Fe1 layer.

For all samples, we have performed the magnetization measurements using a VSM SQUID magnetometer. We have measured the major and minor hysteresis loops  $M(H)$  in order to determine the field range in which the full switching between P and AP orientations of the magnetizations of the Fe1 and Fe2 layers is realized. Figure 1 shows a representative magnetic hysteresis loop for the sample with  $d_{\text{Fe2}} = 0.7\text{ nm}$  which shape is similar for all studied spin valve samples (see also Ref. [13] for details). The pinning of the magnetization of the Fe1 layer by the bias CoO<sub>x</sub> layer was achieved by cooling the sample in a magnetic field of +4 kOe applied parallel to the sample plane down to the base temperature  $T = 4\text{ K}$  at which all hysteresis loops have been measured. The reversal of  $\mathbf{M}_{\text{Fe2}}$  with respect to  $\mathbf{M}_{\text{Fe1}}$  is illustrated by the minor hysteresis loop which was obtained by measuring  $M(H)$  with decreasing the field from +4 kOe down to -1 kOe and increasing the field again up to +1 kOe [Fig. 1(b)]. Notably, the minor loop closes and fully saturates at a field of  $\pm 1\text{ kOe}$  suggesting a complete suppression of the domain state. We find that the height of the loop  $M(\text{P}) - M(\text{AP})$  is proportional to the thickness of the free F2 layer, as expected.

The electrical resistivity measurements were performed with a standard four-point probe setup in the dc mode. The  $T_c$  is defined as the midpoint of the SC transition,

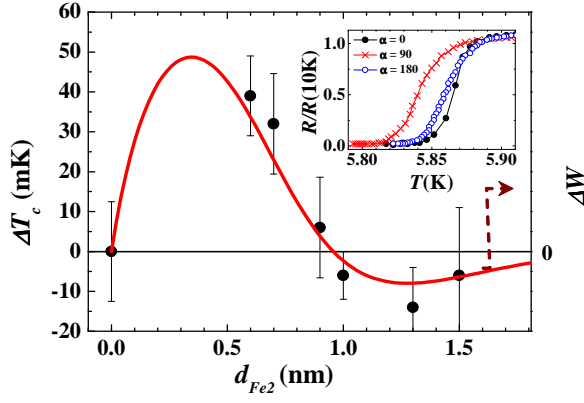


FIG. 2. (Color online) Dependence of the magnitude of the spin valve effect  $\Delta T_c$  on the thickness of the Fe2 layer at a fixed value of the S layer  $d_{\text{Pb}} = 35$  nm. Solid line is a theoretical curve (see the text). Inset shows the SC transition curve for the sample with  $d_{\text{Fe2}} = 1.3$  nm at  $H = 1$  kOe for three different angles between magnetizations of the Fe1 and Fe2 layers.

and the error bars are related with the transition width. We have combined the electrical setup with a vector magnet that enables a continuous rotation of the magnetic field in the plane of the sample. To avoid the occurrence of the unwanted out-of-plane component of the external field the sample plane position was always adjusted with an accuracy better than  $3^\circ$  relative to the direction of the dc external field.

Figure 2 shows the dependence of the magnitude of the spin valve effect  $\Delta T_c = T_c^{\text{AP}} - T_c^{\text{P}}$  on the thickness of the Fe2 layer. For samples with  $d_{\text{Fe2}} < 0.95$  nm, we have observed the direct effect with  $T_c^{\text{P}} < T_c^{\text{AP}}$ , whereas for samples with  $d_{\text{Fe2}} > 0.95$  nm the inverse effect with  $T_c^{\text{P}} > T_c^{\text{AP}}$  has been found. Exemplary SC transition curves for the sample with  $d_{\text{Fe2}} = 1.3$  nm at  $H = 1$  kOe at three different angles  $\alpha$  between  $\mathbf{M}_{\text{Fe1}}$  and  $\mathbf{M}_{\text{Fe2}}$  are shown in the inset to Fig. 2. The data shown in Fig. 2 can be explained within the S/F proximity effect in the framework of the theory of Ref. [5]. In this theory, the set of equations for the singlet SC component in the S layer contains a real-valued parameter  $W(\alpha)$  that enters the boundary condition for the F2/S interface. Physically,  $W(\alpha)$  determines how strongly superconductivity in the S layer is suppressed by the rest of the structure due to the proximity effect. The larger  $W$  is, the stronger  $T_c$  is suppressed. With the notion that  $T_c$  is a monotonic function of  $W(\alpha)$  [5], we plot in Fig. 2 the theoretical result for  $\Delta W = W(0) - W(\pi)$  together with the experimental data on  $\Delta T_c(d_{\text{Fe2}})$  (see Ref. [13] for details). Figure 2 indeed demonstrates a very good correlation between  $\Delta T_c$  and  $\Delta W$  [15]. This proves that the damped oscillating behavior of the spin valve effect is due to the interference of the Cooper-pair wave functions in the Fe2 layer [5, 13].

The central result of our work is an observation in all

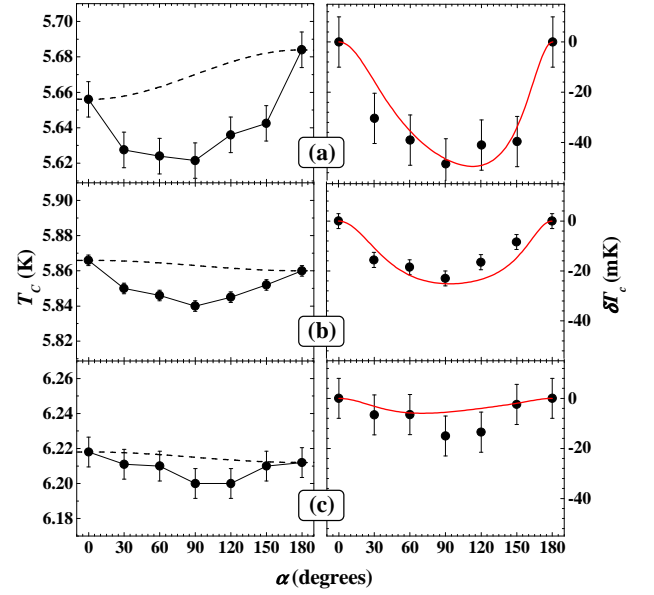


FIG. 3. (Color online) Left: dependence of  $T_c$  on the angle between magnetizations of the Fe1 and Fe2 layers measured in a field  $H = 1$  kOe for the samples with  $d_{\text{Fe2}} = 0.6$  nm (a), 1.0 nm (b), and 1.5 nm (c). Dashed lines are the reference curves calculated according to Eq. (2). Right: deviations  $\delta T_c$  of the actual  $T_c$  values from the respective reference curves. Solid lines are theoretical results for  $\delta W$  (see the text).

spin valve samples of a very special dependence of the SC critical temperature  $T_c$  on the angle  $\alpha$  which the magnetization of the Fe2 layer  $\mathbf{M}_{\text{Fe2}}$  controlled by an external field makes with the magnetization of the pinned Fe1 layer. Examples of such dependences for selected spin-valve samples of different thickness  $d_{\text{Fe2}}$  are shown in Fig. 3. One can see that when changing the mutual orientation of magnetizations by a gradual in-plane rotation of the magnetic field from the P ( $\alpha = 0^\circ$ ) to the AP ( $\alpha = 180^\circ$ ) state,  $T_c$  value does not change monotonically but passes through a minimum. Importantly, for the reference sample consisting of just one Fe layer the angular variation of  $T_c$  lies within the error bars (not shown). In the following, we argue that a characteristic minimum in  $T_c(\alpha)$  close to  $\alpha = 90^\circ$  is a fingerprint of a long-range triplet SC component. Though the triplet component is inherent in the case of noncollinear magnetizations, assuming for a moment its absence one would expect the  $T_c(\alpha)$  dependence to be monotonic. From general symmetry,  $T_c(\alpha)$  must behave as  $\alpha^2$  and  $(\pi - \alpha)^2$  when  $\alpha$  deviates from 0 and  $\pi$ , respectively. One would arrive then at a simple angle-dependent superposition of the limiting values of  $T_c$ :

$$T_c^{\text{(ref)}}(\alpha) = T_c^{\text{P}} \cos^2(\alpha/2) + T_c^{\text{AP}} \sin^2(\alpha/2). \quad (2)$$

This dependence is shown in Figs. 3(a), (b), and (c) (left side) by the dashed lines, and we consider them as reference curves. Deviations  $\delta T_c$  of the actual  $T_c$  from the reference curves are, as the figures demonstrate,

beyond the experimental error bars. The angular dependences of this deviation are shown on the right side panels (a), (b), and (c) of Fig. 3. As discussed above, in the theory of the triplet spin valve in Ref. [5] all information about the magnetic part of the system is encoded into a real positive parameter  $W(\alpha)$ . Hence, the  $\delta T_c(\alpha)$  dependence should correlate with deviation of  $W(\alpha)$  from a reference curve (increase of  $T_c$  corresponds to decrease of  $W$ ); it seems natural to define the reference curve for  $W$  in the same manner as Eq. (2), i.e., as  $W^{(\text{ref})}(\alpha) = W(0) \cos^2(\alpha/2) + W(\pi/2) \sin^2(\alpha/2)$ . The explicit expression for  $\delta W(\alpha)$  can be obtained as described in [5], however, the result is too cumbersome, and we do not present it here. Using the parameters of the theory [17] which we obtained earlier in Ref. [13] and taking the appropriate thicknesses of the Fe2 layer, we find good agreement between the angular dependences of  $T_c$  and  $W$ , see Fig. 3 (right panel).

The dependence of the maximal deviation of  $T_c$ , which we denote as  $\max \delta T_c$ , on the thickness  $d_{\text{Fe2}}$  is shown in Fig. 4. Since  $\max \delta T_c$  occurs near  $\alpha = \pi/2$  the dependence shown in Fig. 4 is close to the dependence of  $\delta T_c(\pi/2) = T_c(\pi/2) - [T_c(0) + T_c(\pi)]/2$  on  $d_{\text{Fe2}}$ . As explained above, this dependence should correlate with  $\delta W(\pi/2) = [W(0) + W(\pi)]/2 - W(\pi/2)$  which in accordance to the theory [5] is equal to

$$\frac{\delta W}{W(0)} = -\frac{\sqrt{2} \sin(2k_h d_{\text{Fe2}} + \pi/4) - e^{-2k_h d_{\text{Fe2}}}}{2 [\sinh(2k_h d_{\text{Fe2}}) + \cos(2k_h d_{\text{Fe2}})]} - \frac{4 [\sin^2(k_h d_{\text{Fe2}}) - 2k_\omega d_{\text{Fe2}}]}{e^{2k_h d_{\text{Fe2}}} - 2 + \sqrt{2} \cos(2k_h d_{\text{Fe2}} + \pi/4) + 4k_\omega/k_h}. \quad (3)$$

Here,  $k_\omega = \sqrt{2\omega/D}$ ,  $k_h = \sqrt{h/D}$ ,  $\omega = \pi T_c(2n+1)$  with integer  $n$  is the Matsubara frequency,  $h$  is the exchange field in the Fe2 layer along the  $z$  direction and  $D$  is the diffusion coefficient of conduction electrons in the F layer. The result (3) is valid at  $k_\omega \ll k_h$  (obviously satisfied in real ferromagnets with  $T_c \ll h$ ),  $k_\omega d_{\text{Fe2}} \ll 1$  (that is the limit in which the triplet component is maximal since its spatial damping is negligible),  $k_\omega d_S \gtrsim 1$  (this means that the S layer is not too thin and hence the superconductivity is preserved). The curve corresponding to Eq. (3) demonstrates good agreement with the experimental results (Fig. 4) confirming that the increase/decrease of  $T_c$  indeed correlates with the decrease/increase of  $W$ . This model curve has been obtained with the same set of parameters as the ones in Fig. 3 [17]. Bearing in mind that the effect is not observed for the reference sample  $\text{CoO}_x/\text{Cu}/\text{Fe}/\text{Pb}$  with a single iron layer we interpret our finding as evidence for long-range triplet superconductivity that arises in the spin-valve samples with a non-collinear geometry of magnetizations of the Fe1 and Fe2 layers.

Finally, we mention that earlier indications for long-range superconductivity in an F layer have been detected through the proximity-induced conductance [18, 19] even

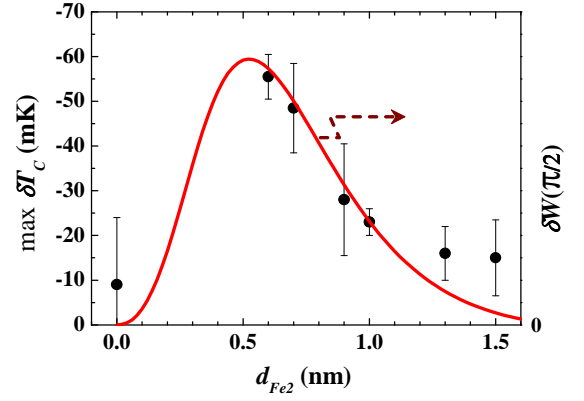


FIG. 4. (Color online) Dependence of the maximal deviation of  $T_c$ ,  $\max \delta T_c$ , on the thickness of the Fe2 layer. Solid line is the theoretical result for  $\delta W(\pi/2)$  according to Eq. (3) (see the text).

before the theoretical works have appeared. Recently, the occurrence of the odd in the Matsubara frequency triplet superconductivity in the S/F/S systems, predicted in Ref. [6], was inferred from the experiments on Josephson junctions through observation of the anomalously deep penetration of the Cooper condensate into the F layer (see, e.g., [20–26]). We note that our experiments are advantageous in that they address the primary SC parameter of the spin valve, the behavior of  $T_c$ , which is directly affected by the spin-triplet component.

In conclusion, we have studied the behavior of the superconducting critical temperature  $T_c$  of the S/F spin valve system  $\text{CoO}_x/\text{Fe1}/\text{Cu}/\text{Fe2}/\text{Pb}$  with different thicknesses of the ferromagnetic Fe2 layer. We have observed a remarkable nonmonotonic dependence of  $T_c$  on the angle between the directions of the magnetization  $\mathbf{M}$  in the Fe1 and Fe2 layers. The  $T_c$  passes through a clear minimum near the orthogonal orientation of  $\mathbf{M}_{\text{Fe1}}$  and  $\mathbf{M}_{\text{Fe2}}$  which is not expected in the case of singlet superconductivity. We argue that this particularly strong suppression of  $T_c$  in the orthogonal geometry is due to an enhanced “leakage” of the SC Cooper pairs into the F layer occurring via the long-range spin-triplet channel.

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